

This Page Is Inserted by IFW Operations  
and is not a part of the Official Record

## **BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning documents *will not* correct images,  
please do not report the images to the  
Image Problem Mailbox.**

**THIS PAGE BLANK (USPTO)**

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION  
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification <sup>7</sup> :</b> <b>A61L 15/58, A61F 13/15, 5/44</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 00/45865</b> <b>(43) International Publication Date:</b> 10 August 2000 (10.08.00)
<b>(21) International Application Number:</b> PCT/US00/02706 <b>(22) International Filing Date:</b> 2 February 2000 (02.02.00) <b>(30) Priority Data:</b> 99102048.8 2 February 1999 (02.02.99) EP <b>(71) Applicant (for all designated States except US):</b> THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US). <b>(72) Inventors; and</b> <b>(75) Inventors/Applicants (for US only):</b> CINELLI, Fabio [IT/IT]; Via Duccio da Boninsegna, 8, I-40133 Bologna (IT). COLAIANNI, Antonello [IT/IT]; Via Enrico Jasonni, 16, I-65126 Pescara (IT). TORDONE, Adelia, Alessandra [IT/IT]; Via Monte Rotondo, 26, I-65125 Pescara (IT). MUNRO, Hugh, Semple [GB/GB]; Barton Cottage, Weston Sub Edge, Chipping Camden (GB). TIGHE, Brian, John [GB/GB]; Aston Triangle, Birmingham B47ET (GB). <b>(74) Agents:</b> REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217-1087 (US).		<b>(81) Designated States:</b> AE, AL, AM, AT, AT (Utility model), AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, CZ (Utility model), DE, DE (Utility model), DK, DK (Utility model), DM, EE, EE (Utility model), ES, FI, FI (Utility model), GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SK (Utility model), SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> DISPOSABLE ABSORBENT ARTICLES WITH WATER ADHESION STABILITY <b>(57) Abstract</b> <p>The present invention relates to disposable absorbent articles such as diapers and sanitary napkins which are provided with adhesives for attachment of the article to the skin. In particular the present invention relates to adhesives which provide secure attachment and are pleasing to the skin upon application, yet cause no discomfort upon removal. In particular the present invention relates to an adhesive which provides secure attachment under moist and wet skin conditions and which maintains adhesive peel strength even under exposure to excess water.</p> <p><i>Published after priority date</i></p>		

**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece			TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Malawi	US	United States of America
CA	Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	NZ	New Zealand		
CM	Cameroon			PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
CU	Cuba	KZ	Kazakhstan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Estonia	LR	Liberia	SG	Singapore		

## DISPOSABLE ABSORBENT ARTICLES WITH WATER ADHESION STABILITY

5

10

15

Field of the Invention

20

25

The present invention relates to a disposable absorbent articles such as diapers, sanitary napkins, pantliners, tampons, perspiration pads, adult incontinence devices and the like to be attached directly to the skin of the wearer. The articles utilise an improved adhesive so as to facilitate easy application and removal of the article from the wearer, whilst ensuring maintenance of the article in the desired position. In particular the adhesives provide attachment on moist and wet skin for the entire period of wear, including circumstances or periods of wear during which the adhesive is exposed to excess amounts of liquids.

Background of the Invention

30

35

The present invention relates to adhesives which are particularly useful to absorbent articles for absorption of body liquids which naturally emanate from a body without a wound. For example to attach sanitary napkins or pantliners in the genital region. Also incontinence devices which are worn e.g. in the genital region or perspiration pads which are worn in the arm pit region of a person can suitably employ the adhesive of the present invention.

Such adhesives have been generally disclosed in for example US statutory invention registration H1602 or WO 96/33683 and WO 95/16424. The latter discloses sanitary articles having a topical adhesive which is applied on the wearer facing side of a sanitary napkin along the entire periphery. WO 96/13238 discloses a topical adhesive which is described in terms of frequency dependency. EP 638 303 discloses the use of a topical adhesive on side cuffs of sanitary napkins in order to keep the cuffs in an upright position. Swiss publication CH-643730 discloses the use of a very long sanitary napkin having chamfered outer edges with a topical adhesive at the four corners of the outer edges in order to provide a topical adhesive area well outside the region of pubic hair growth.

However all of these disclosures typically disclose a product which is designed to be utilised in combination with an undergarment and hence the degree of adhesion actually provided is very low and is not designed to withstand any excessive pressure. Moreover the adhesive is only discussed in general terms or concentrates on the area of application of the adhesive to the article. The nature of adhesive per se other than the basic physical requirements such as pressure sensitivity are not discussed in particular with reference to the chemical composition or the adhesive criteria.

The prior art in the general field of adhesives for attachment to the skin is particularly developed in the field of articles such as band-aids, plasters and bandages. These articles are however typically applied in an emergency situation, where for example, a cut into the skin of the wearer has occurred and absorption of the body liquids emanating from a wound is desired. In this context performance aspects of the article such as easy application and use of the product, comfortable wear as well as painless removal, and discreteness are again subordinate, to other criteria in this case such as sterility, healing support, and mechanical protection of the wound. Also such wound covering absorbent articles are mostly adhered to the skin where prior to application of the absorbent article bodily hair can be removed or where little hair grows.

In order to provide the desired level of adhesion of such bandages, the prior art typically discloses the utilisation of certain adhesives having very high cohesive strengths such as rubber based adhesives and acrylics. These

adhesives are then applied as thick layers to maximise the adhesive force by which the bandage is secured to the skin of the wearer.

5 US 4 699 146 discloses hydrophilic elastomeric pressure sensitive adhesives suitable for use with ostomy devices, bandages, ulcer pads, sanitary napkins, diapers, and althetic padding. The adhesive comprises at least 1 uradiation cross linked organic polymer and an adhesive plasticizer.

10 GB 2 115 431 discloses adhesives for bandages, wounds or burn dressings, EKG adhesives, sanitary napkins, diapers and ulcer pads. The adhesive comprises an uradiation cross linked organic polymer such as polyvinylpyrrolodine and an adhesive plasticizer.

15 However, for application such as absorbent articles it is important that the adhesive has a skin compatible composition and not be harsh or aggressive towards the skin or cause skin irritation or inflammation. Also it is preferred if the adhesive is compliant with the skin of the wearer such that maximum skin surface contact between the adhesive and the skin is achieved. Moreover, it is also desirable to provide an adhesive such that the absorbent article can be  
20 readily removed from the wearer, without the wearer experiencing any unacceptable pain level. This is particularly important under circumstances, where the article is removed and reapplication of the article once or even a number of times is required for example to allow for urination and or to ensure the application of such articles on sensitive skin and wearer groups such as  
25 infants. However, on the other hand the desired level of adhesion, albeit painless should of course also be maintained during such multiple applications of the article.

30 The problem of achieving the desired adhesion level is further exacerbated under wet skin conditions. Typically, prior to the placement of the article the skin is cleaned and is usually as a result moist. The currently available adhesives, such as hydrocolloids, however often do not immediately strongly adhere to the skin and may need to be held in place until sufficient minimum adhesion occurs. Moreover, the overall adhesive ability of such adhesives tends to be significantly  
35 reduced on wet skin surfaces per se, so that the article will typically not remain

attached to the skin during wear if any pressure is exerted onto the article, for example by the movement of the wearer.

Moist and wet skin however is not just a problem which is prevalent at the article application stage as a significant amount of moisture is also generated during the use of the article from the wearer by perspiration and from bodily fluids. Under such circumstances currently available adhesives typically cannot absorb this moisture and again the adhesive strength is reduced to such an extent that the article will often become detached under exertion of pressure during wear. It is hence very important to provide an adhesive which provides both initial adhesion and maintenance of its adhesive strength on wet skin. Moreover, it is also another important factor for the product performance that the adhesive is also stable to exposure to excess quantities of liquid such as water, urine and menstrual fluids and will also not lose its adhesive strength under such circumstances.

None of the prior art in the field of absorbent articles however even recognises or addresses the problem of providing these articles with an adhesive which meets these criteria, in particular adhesives which adhere to wet skin and are stable and maintain their adhesiveness even when exposed to excessive amounts of liquid.

Adhesion to wet skin is addressed for example in WO 98/03208 which discloses medical pressure sensitive adhesives which can adhere to dry or wet skin and which comprise a mixture of hydrophilic (meth)acrylate copolymer containing tertiaryamino groups, a hydrophilic (meth)acrylate copolymer containing carboxyl groups, carboxylic acids and a crosslinking system. However this document does not discuss adhesion after exposure to excess liquid.

Hence there still exists a need to disposable absorbent articles having an adhesive for the secure attachment and painless removal of the article from the skin suitable for use of sensitive skin of an infant and or of the genitalia and it is thus an object of the present invention to provide such an article.

It is another objective of the present invention to provide an adhesive that exhibits an ability to adhere to skin upon reapplication, particularly multiple



reapplication for example when the article is removed for urination purposes or is misplaced, whilst still allowing painless removal.

5 It is yet a further objective of the present invention that the adhesive will adhere to moist or wet skin, independent of whether this is direct application of the article onto wet skin, or moisture which is generated on the skin surface during the wearing period of the article. In particular it is an objective of the present invention to provide an adhesive which is liquid stable particularly to water and urine, such that the adhesion properties will not be significantly  
10 effected in the presence thereof over the period of wear of the article.

It is another object of the present invention to provide an adhesive which upon removal from the skin of the wearer leaves no residues. It is yet another object of the present invention to provide an adhesive which does not cause a  
15 cold or otherwise unacceptable temperature sensation upon application to the wearer.

An additional object of the present invention to provide an adhesive which provides flexibility, stretchability and contractability so that it is able to adapt to  
20 the contours of the body during all bodily movements and hence be comfortable for the wearer of the article, whilst still having sufficient adhesive capacity to ensure secure attachment during use.

It has now been surprisingly found that the above drawbacks will be  
25 substantially alleviated by providing the absorbent article with an adhesive as defined hereinafter. The adhesive provides secure attachment, is pleasing to the skin upon application, and yet causes no discomfort upon removal and maintains its adhesive strength over the period of wear even under exposure to excess liquids.

30

#### Summary of the Invention

Any disposable absorbent article known in the art can be provided with the adhesive according to the present invention. According to the present invention  
35 the adhesive is provided so as to have an initial peel strength ( $P_i$ ) under normal ambient conditions and a final peel strength ( $P_f$ ) after exposure to water

according to the test method described herein, whereby the ratio of  $P_i$  to  $P_F$  is from 2:1 to 2:4 preferably from 2:1.25 to 2:2.5 and has a water absorption capacity as defined in the test herein of at least 3% by weight of said adhesive..

5       The adhesive allows attachment of disposal absorbent articles to the skin of the wearer, the adhesive being provided as a layer having a certain thickness or calliper C measured in millimetres (mm), typically on at least part of the wearer facing surface of the article.

10       Detailed analysis of the sequence of common situations occurring from the application of absorbent articles to the time of removal of such articles has shown that specific adhesive characteristics need to be preferably satisfied in order to achieve the desired performance objectives, in particular to secure initial attachment, secure attachment during use and painless removal after wear. The  
15       characteristics which have been considered in this context are the elastic modulus describing the elastic behaviour of the material and the viscous modulus which describes the viscous behaviour of the adhesive material.

20       The viscous behaviour of the adhesive can be interpreted to represent an indication of the ability of the adhesive to quickly attach and securely adhere to a particular surface. The elastic behaviour can be interpreted as an indication of the "hardness" behaviour of the adhesive. Its value is also important for good initial attachment. Their combination is believed to be an indicator of the required force upon removal. The relation between elastic and viscous modulus is  
25       considered to be an indication on which fraction of the removal energy will be dissipated within the adhesive and which fraction is available to trigger the actual removal.

30       In order to provide adhesives for secure initial and prolonged attachment and easy/painless removal the relation between the elastic modulus and the viscous modulus as well as their dynamic behaviour is also of importance.

35       The adhesive has an elastic modulus at a temperature of 37°C (100° Fahrenheit) abbreviated  $G'_{37}$ , a viscous modulus at a temperature of 37°C (100° Fahrenheit) of  $G''_{37}$ , and a viscous modulus at a temperature of 25°C (77°Fahrenheit) of  $G''_{25}$ .

The adhesive according to the present invention preferably satisfies the following conditions;

5             $G'_{37}$  (1 rad/sec)            is in the range 500 Pa to 20000 Pa,  
   preferably 700 Pa to 15000 Pa, most  
   preferably 1000 Pa to 10000 Pa.

10             $G''_{37}$  (1 rad/sec)            is in the range 100 Pa to 15000 Pa,  
   preferably 100 Pa to 10000 Pa, most  
   preferably 300 Pa to 5000 Pa.

                 and the ratio of  $G'_{37}$  (1 rad/sec) /  $G''_{37}$  (1 rad/sec) is in the  
   range of 1 to 30.

15

                 Provided the above rheological conditions are satisfied the adhesives will  
                 also satisfy conditions such as sufficient cohesiveness (to prevent residue of  
                 adhesive on the skin) which are important for commercial use of such adhesives  
                 and apparent to those skilled in the art. Adhesive compositions which satisfy the  
20            above criteria can be used as adhesives for the article provided they also satisfy  
                 the common requirements of being safe for use on human or animal skin during  
                 use and generally after disposal of the article.

25            Often the criteria of hygienic appearance such that adhesive compositions  
                 which are transparent or white upon application are preferred.

                 It has been determined that the relation between the thickness or calliper C,  
                 measured in millimetres (mm), of the layer in which the adhesive is provided,  
                 typically onto at least a portion of the wearer facing surface of the article, and the  
30            viscous modulus  $G''_{25}$  at about 100 rad/sec of the adhesive, is relevant to the  
                 scope of providing an easy and painless removal from the wearer's skin of such  
                 a adhesive applied on at least a portion of the wearer facing surface of an  
                 absorbent article for attachment of said article to the skin of a wearer.

The adhesive of the present invention is thus preferably provided as a layer having a thickness C such that the viscous modulus  $G''_{25}$  (100 rad/sec) and the thickness C preferably satisfy the following empirical equation:

5 
$$G''_{25} \leq [(7.00 + C) \times 3000] \text{ Pa}$$

and preferably also the following empirical equation:

10 
$$G''_{25} \leq [(5.50 + C) \times 1700] \text{ Pa}$$

#### Detailed Description of the Invention

15 According to the present invention the adhesive can be utilised on disposable absorbent articles such as diapers, sanitary napkins, panty liners, incontinence devices, perspiration pads and tampons. The word "skin" according to the present invention does not only relate to the specific derma of the user but includes the mucous tissue as well as the hair which is typically found in the genital region.

20 Due to the nature and environment in which such disposable absorbent articles are utilised it is an essential feature that the adhesive has a water absorption capacity as defined in the test herein of at least 3% by weight of said adhesive (so that the adhesive adheres directly onto wet or moist skin). In addition it is also essential that the adhesive maintains its adhesive strength in the presence of excess liquid. In particular, the ratio of the peel strength of the adhesive as determined in the test methods herein should most preferably be maintained at a constant value such that the ratio of initial peel strength ( $P_i$ ) and the final peel strength ( $P_f$ ) is from 2.0:1 to 2:4, preferably from 2:1.25 to 2:2.50, most preferably from 2.0:1.5 to 2.0:2.5. Typically for disposable absorbent articles the initial peel strength for dry and more preferably also for wet skin should be from 0.1N/cm to 5.0N/cm, preferably from 0.5N/cm to 3.0N/cm.

35 It is further also preferable that the adhesive in addition to maintaining its peel strength over a period of time even in the presence of water also absorbs less than 15 %, preferably less than 10%, more preferably less than 7% water. Whilst not intending to being bound by theory, it is believed that in order to obtain

direct adhesion onto wet skin and maintain constant adhesion performance over a period of wear, even when exposed to excess liquids or high humidity the ability of the adhesive to absorb water needs to be considered. In particular, it has been identified that, not only the absolute ability of the adhesive needs to be considered, but also the rate of water absorption in order to provide an adhesive meeting the above identified performance parameters.

For example hydrocolloid adhesives which are known in the art comprising a 3-dimensional rubber matrix and colloidal absorbent particles dispersed therein are only able to absorb limited amounts of water through the colloidal particles themselves and not the matrix itself. In addition the rate at which water is absorbent is slow. Hence these prior art adhesives do not adhere to wet surfaces.

Prior art hydrogel adhesives on the other hand are able to not only absorb large quantities of water but also at a very fast rate. As a result such adhesives may be able to adhere, to wet surfaces, however due to the combination of fast rate of absorption and large absolute water uptake, these adhesives loose their adhesive strength rapidly in the presence of excess water or high humidity.

Accordingly the adhesives of the present invention exhibit both an ability to adhere directly to wet skin, by having a minimum absolute water absorption ability in combination with a rate of absorption such that the peel strength remains within defined levels over the period of wear.

The adhesive is provided with the preferred pattern, typically on the wearer facing surface of the article, as a layer having a thickness or calliper C that is preferably constant. The layer can be preferably continuous or alternatively discontinuous, e.g. in form of dots, spirals, or stripes.

Even though adhesives are used like pressure sensitive adhesives on human skin hair and mucous tissues, it is understood that the topical adhesive compositions could only with difficulty be considered typical pressure sensitive adhesives (referred to as PSA hereinafter) on the basis of the most characteristic rheological behaviours identifying such materials.

In fact as the person skilled in the art of adhesives knows, the most characteristic feature that distinguishes a PSA from other substances that can temporarily adhere objects (e.g. water between two glass plates could) is the fact that their rheological parameters and especially the Elastic Modulus  $G'$  vary greatly with the frequency of applied stresses. More in particular,  $G'$  of PSA can increase over some orders of magnitude, while the frequency of applied stresses varies from typical bonding frequency to typical debonding frequency, i.e. 1 rad/s to 100 rad/s as indicated below.

As a first consequence, it is therefore inadmissible to define materials intended for use as "adhesives" by giving values of rheological parameters and especially of  $G'$  at a fixed value of frequency. This can be misleading because in the absence of other characteristics it will include materials which have no practical value. It is hence necessary that rheological characterisation must be on the basis of dynamic considerations. This not only applies to the Elastic Modulus  $G'$  but also to the viscous modulus  $G''$  and hence also for  $\tan(\delta) = G'' / G'$ .

It is well known that typical PSAs have not only a high variation of  $G'$  across the considered frequencies, but also that there is an even higher variation of  $G''$  which can get close or become even higher than the value of  $G'$ , i.e.  $\tan(\delta)$  becomes about or even greater than 1, in particular at the frequencies that are typical of debonding.

Without wishing to be bound by theory this can be interpreted as meaning that a high fraction of the energy applied for the debonding is dissipated within the adhesive (so it is not effective in causing the debonding) and through the interface of the adhesive and the skin, while this fact causes macroscopically the recording of a very high level of adhesive force.

As indicated above materials useful as adhesives according to the present invention have rheological characteristics which are measured at a reference temperature of 37°C (as usual body temperature of humans) and in a range of frequencies. It has been found that upon application of a disposable absorbent article with a adhesive the adhesive contact is formed at a low frequency, while debonding happens at the speed of removing the article. This speed is expressed as a frequency of 100 rad/s, while the low frequency of forming the

adhesive bond has been found to be on the order of 1 rad/s. Therefore, the frequency range for use according to the present invention is between 1 and 100 rad/s.

5 In order to provide good conditions of bonding, i.e. at a frequency of about 1 rad/sec, the absolute values of the elastic modulus should not be too high, otherwise the adhesive is too hard and it is not able to intimately join or mold to the surface to which it is expected to adhere. It is also important to have a low absolute value of  $G''$  in order to have good cohesion while the material remains  
10 soft and capable of gently adhering to skin.

The ratio of  $G'_{37}$  (1 rad/sec) over  $G''_{37}$  (1 rad/sec) is important to ensure that these two values are balanced upon adhesion to the skin.

15 Importantly, the ratio of  $G'_{37}$  (100 rad/sec) -  $G''_{37}$  (100 rad/sec)

$$\frac{G'_{37} (100 \text{ rad/sec}) - G''_{37} (100 \text{ rad/sec})}{G'_{37} (1 \text{ rad/sec}) - G''_{37} (1 \text{ rad/sec})}$$

20 needs to be large enough to ensure that the dynamic behaviour of both the elastic and the viscous module are maintained in a relationship which provides secure adhesion and painless and easy removal.

25 Finally the person skilled in the art will also recognise that the Glass Transition Temperature  $T_g$  of the adhesive composition, the specific heat capacity, and the specific heat conductivity are parameters which are useful to more fully define the group of useful adhesives.

30 The following set of characteristics should preferably be satisfied for the adhesive of the present invention:

$G'_{37}$  (1 rad/sec) is in the range 500 Pa to 20000 Pa,  
preferably 700 Pa to 15000 Pa, most  
preferably 1000 Pa to 10000 Pa.

35  $G''_{37}$  (1 rad/sec) is in the range 100 Pa to 15000 Pa,  
preferably 100 Pa to 10000 Pa, most

preferably 300 Pa to 5000 Pa.

the ratio of  $G'_{37}$  (1 rad/sec) /  $G''_{37}$  (1 rad/sec)  
is in the range of 1 to 30.

the ratio 
$$\frac{G'_{37} (100 \text{ rad/sec}) - G''_{37} (100 \text{ rad/sec})}{G'_{37} (1 \text{ rad/sec}) - G''_{37} (1 \text{ rad/sec})}$$

is not less than 0.5, preferably in the range 0.7  
to 3, most preferably in the range 1 to 1.8.

The value of the ratio of  $G'_{37}/G''_{37}$  at least for the frequency range above 1  
rads/up to 100 rads/s should preferably be not less than 0.5, preferably from 0.7  
to 10 and most preferably from 1 to 7.

The rheological behaviour can also be related to the values of the Glass  
Transition Temperature  $T_g$ . For topical adhesives according to the present  
invention  $T_g$  should preferably be less than  $0^\circ\text{C}$ , more preferably less than  $-5^\circ\text{C}$   
and most preferably less than  $-10^\circ\text{C}$ .

In order to provide adhesive compositions which satisfy the requirements of  
the above rheological and physical characteristics of an adhesive any medically  
suitable substantially water insoluble pressure sensitive adhesives comprising a  
polymer which forms a 3-dimensional matrix meeting the these characteristics  
may be utilised.

According to the present invention the 3-dimensional matrix also referred to  
herein as a gel, comprises as an essential component a polymer which can be  
physically or chemically cross linked. The polymer may be naturally or  
synthetically derived. The uncrosslinked polymer includes repeating units or  
monomers derived from vinyl alcohols, vinyl ethers and their copolymers, carboxy  
vinyl monomer, vinyl ester monomers, esters of carboxy vinyl monomers, vinyl  
amide monomers, anionic vinyl monomers, hydroxy vinyl monomers, cationic  
vinyl monomers containing amines or quaternary groups, N-vinyl lactam  
monomer, polyethylene oxides, polyvinylpyrrolidone (PVP), polyurethanes,  
acrylics such as methyl acrylate, 2-hydroxyethyl methacrylate,



methoxydiethoxyethyl methacrylate and hydroxydiethoxyethyl methacrylate, acrylamides, and sulphonated polymers such as acrylamide sulphonated polymers for example 2 acrylamido methylpropane sulphonic acid (AMPs) and acrylic (3-sulphopropyl) ester acid (SPA), and mixtures thereof. Also acrylonitrile, methacrylamide, N,N-dimethylacrylamide (NNDMA), acrylic esters such as methyl, ethyl and butyl acrylates. Alternatively, the uncrosslinked polymer may be a homopolymer or copolymer of a polyvinyl ether, or a copolymer derived from a half ester of maleic ester. Similarly any other compatible polymer monomer units may be used as copolymers such as for example polyvinyl alcohol and polyacrylic acid or ethylene and vinyl acetate.

As another alternative, the polymers may be block copolymer thermoplastic elastomers such as ABA block copolymers such as styrene-olefin-styrene block copolymers or ethylene-propylene block copolymers. More preferably such polymers include hydrogenated grade styrol/ethylene-butylene/styrol (SEBS), styrene/isoprene/styrene (SIS), and styrol/ethylene-propylene/styrol (SEPS).

Particularly preferred polymers are acrylics, sulphonated polymers such as acrylamide sulphonated polymers, vinyl alcohols, vinyl pyrrolidone, polyethylene oxide and mixtures thereof. Most preferred are nitrogen containing polymers.

According to the present invention the 3-dimensional adhesive matrix also essentially comprises a plasticiser, which is preferably a liquid at room temperature. This material is selected such that the polymer may be solubilized or dispersed within the plasticiser. For embodiments wherein irradiation cross linking is to be carried out, the plasticiser must also be irradiation cross linking compatible such that it does not inhibit the irradiation cross linking process of the polymer. The plasticiser may be hydrophilic or hydrophobic.

Suitable plasticisers include water, alcohols, polyhydric alcohols such as glycerol and sorbitol, and glycols and ether glycols such as mono- or diethers of polyalkylene glycol, mono- or diester polyalkylene glycols, polyethylene glycols (typically up to a molecular weight of about 600), glycolates, glycerol, sorbitan esters, esters of citric and tartaric acid, imidazoline derived amphoteric surfactants, lactams, amides, polyamides, quaternary ammonium compounds, esters such as phthalates, adipates, stearates, palmitates, sebacates, or myristates,

and combinations thereof. Particularly preferred are polyhydric alcohols, polyethylene glycol (with a molecular weight up to about 600), glycerol, sorbitol, water and mixtures thereof.

5 Typically the adhesive comprises a ratio of polymer to plasticiser by weight of from 1:100 to 100:1, more preferably from 50:1 to 1:50. However, the exact amounts and ratios of the polymer and plasticiser will depend to a large extent on the exact nature of polymer and plasticisers utilised and can be readily selected by the skilled person in the art. For example a high molecular weight polymer  
10 material will require a greater amount of plasticiser than a low molecular weight polymer.

Other common additives known in the art such as preservatives, antioxidants, pigments, mineral fillers and mixtures thereof may also be  
15 comprised within the adhesive composition in quantities up to 10 % by weight each respectively.

According to the present invention the polymer component of the adhesive can be physically or chemically cross linked in order to form the 3 dimensional  
20 matrix. Physical cross linking refers to polymers having cross links which are not chemical covalent bonds but are of a physical nature such that there are areas in the 3 dimensional matrix having high crystallinity or areas having a high glass transition temperature. Chemical cross linking refers to polymers which are linked by chemical bonds. Preferably the polymer is chemically cross linked by radiation  
25 techniques such as thermal-, E beam-, UV-, gamma or micro-wave radiation.

In addition when chemical crosslinks are formed in the system, a polyfunctional cross linker and/or a free radical initiator may be present in the premix to initiate the crosslinking upon irradiation. Such an initiator can be  
30 present in quantities up to 5% by weight, preferably from 0.02% to 2%, more preferably from 0.02% to 0.2%. Suitable photoinitiators include type I- $\alpha$ -hydroxyketones and benzilidimethyl-ketals e.g. Irgacure 651 which are believed to on irradiation to form benzoyl radicals that initiate polymerization. Photoinitiators of this type that are preferred do not carry substituents in the para position of the aromatic ring. Particularly preferred is 1-hydroxycyclohexylphenylketone  
35 (available under the trade name Irgacure 184 from Ciba Sp ciality Chemicals)

also preferred are Darocur 1173 (2-hydroxy-2-propyl phenyl ketone) and mixtures of Irgacure 184 and Darocur 1173. In addition from 0.02% to 2% of thermal initiators may also be used.

5       The resulting adhesive compositions is mainly hydrophilic. Hydrophobic and mixed phase compositions are dependant upon the nature of the components of the adhesive. In addition a mixture of monomers whether hydrophilic or both hydrophobic and hydrophilic may result in a single phase or mixed phase of at least 2 phases. Preferably, the adhesives of the present invention are mixed  
10   phase hydrophilic hydrophobic.

A mixture of monomers which may result in 1, 2 or more phases are preferred. Mixed phase adhesives are compositions in which both hydrophobic and hydrophilic components, preferably in both plasticisers and polymers, form  
15   two or more separate phases. In such cases an emulsifier is preferably present at a suitable level to form stable emulsions between the incompatible phases.

Whilst not intending to be bound by theory it is believed that the improved peel strength liquid stability particularly with respect to water of the adhesives is  
20   obtained from a monomer mix comprising both hydrophilic e.g. polar and/or ionic monomers, preferably ionic water soluble monomer and hydrophobic i.e water insoluble monomers. Preferably the ratio of hydrophilic monomers to hydrophobic monomers should be in the range of from 5:1 to 1:5 and preferably from 3:1 to 1:3, preferably from 2:1 to 1:2. The hydrophilicity and hydrophobicity of a  
25   monomer component is always relative to the other component. Typically prior art hydrogel adhesives comprise hydrophilic monomers only, as a consequence of which they have a high rate of water absorption and do not maintain adhesion after exposure to excess liquid. Whilst not intending to be bound by theory, it is believed that the presence of a hydrophobic component in the adhesive matrix  
30   reduces the rate of absorption of water of the adhesive. As a result the distribution of the water absorbed by the adhesive is more uniform. Consequently a water film is not generated between the surface of the skin and the adhesive, which if present, prevents the formation of bonds between skin and adhesive and thus th adhesive capacity of the adhesive itself.

Thus the invention seeks to provide a homogeneously dispersed reaction mixture comprising both hydrophobic and hydrophilic components which, on polymerisation separates into a biphasic or a multiphasic structure. The phases have in some cases been observed to have a thickness of about 100 microns +/- 50 microns. The reaction mixture may contain one or more surface active agents which may assist or promote phase separation but in the course of polymerisation become anisotropically distributed between the result phases.

The presence of a hydrophobic monomer or polymer may be necessary in the initial homogenous dispersion in order to more effectively promote phase separation.

Suitable preferred hydrophilic monomers are acrylic acid, and salts thereof, 2-acrylamido methylpropane sulphonic acid, acrylic (3-sulphopropyl) ester acid and salts thereof and combinations thereof. A particularly preferred example is 2-acrylamide-2-methylpropane sulphonic acid sodium salt, commonly known as NaAMPs, available commercially from Lubrizol as either a 50% aqueous solution (reference code LZ 2405) or at a 58% aqueous solution (reference code LZ 2450 A). Suitable hydrophobic monomer components are methyl-, ethyl-, butyl hexyl, iso octyl- and isodecyl acrylates and methacrylate, vinyl ethers, vinyl pyrrolidine, glycidyl acrylate and 2-ethoxyethyl acrylate, tetra-hydrofurfuryl acrylate, hydroxypropyl acrylate, vinyl propionate and vinyl butyrate, and combinations thereof. Particularly preferred are ethoxy ethyl acrylate or butyl acrylate.

When the adhesive comprises a hydrophobic component, such as butyl acrylate as well as a hydrophilic monomer (i.e. the aforesaid water soluble ionic monomer), such as NaAMPS, the presence of a nonionic water soluble monomer, for example NNDMA, is preferred to act as a so-called "reactive solvent bridge" to provide intimate mixing of the various seemingly incompatible components of the reaction mixture prior to polymerisation. The reaction mixture thus has a homogenous structure containing both hydrophilic and hydrophobic components that are intimately mixed, as the NNDMA acts as a solvent for both hydrophilic and hydrophobic materials, providing a clear compatible coating solution or dispersion. As the reactive solvent bridge is polymerised and thus essentially removed from the reaction mixture the stability of the system is

adversely affected and the compatible coating solutions or dispersions undergo phase separation so as to provide a biphasic structure.

In a preferred embodiment of the invention the aforesaid non ionic water soluble monomer will comprise at least one of a mono- or di-N-alkylacrylamide or an analogue thereof. The term "analogue" in this context refers to non ionic water soluble monomers containing an alkyl or substituted alkyl group linked to a carbon-carbon double bond via an amido or alkylamido (-CO.NH- or CO.NR-) function. Examples of such analogues include diacetone acrylamide (N-1,1-dimethyl-3-oxobutyl-acrylamide), N-alkylated acrylamides, N,N-dialkylated acrylamides, N-vinyl pyrrolidone and acryloyl morpholine. N,N-dimethylacrylamide (NNDMA) and/or and analogue thereof is preferred. The reaction mixture preferably comprises from about 15% to about 30% and ideally from about 15% to about 25%, by weight of the reaction mixture, of the non ionic water soluble monomer.

The term "reactive solvent bridge" used herein refers to a partially lipophilic non ionic water soluble monomer which has the ability to partition between the hydrophobic and aqueous phases, whereby the hydrophobic monomer is substantially solubilised in the homogeneous reaction mixture before polymerisation begins. The solvent bridge is reactive in that it is a polymerisable monomer which takes part in the polymerisation reaction. Without wishing to be bound by theory, it is believed that the solvent bridge function of the non ionic water soluble monomer is exercised predominantly prior to, and in the relatively early stages of, the polymerisation reaction, and reduces as the polymerisation reaction proceeds.

In certain circumstances the reaction mixture preferably comprises from 3% to 20%, and more preferably from 8% to 18% by weight of the reaction mixture, of a stabilised polymer dispersion that is used to provide a stable phase separated system. The polymer preferably comprises any of the following either alone or in combination: vinylacetate dioctyl maleate copolymer or ethylene-vinyl acetate copolymer. Ethylene-vinylacetate copolymer is preferred, such as that marketed under the trade name DM137 by Harlow Chemicals.

The adhesive is thus typically formed by polymerising a homogeneous aqueous reaction comprising from 5 to 50%, preferably from 30% to 50% by weight of the reaction mixture, of hydrophilic monomer, i.e. an ionic water soluble monomer, from 10% to 50%, preferably from 15% to 45% by weight of the reaction mixture, of a plasticiser (other than water), up to 50%, preferably from 10% to 50%, more preferably from 15% to 30%, most preferably from 15% to 25% by weight of the reaction mixture, of a hydrophobic nonionic monomer, i.e. nonionic water soluble monomer, from up to 40%, preferably from 0.05% to 40%, more preferably from 3 to 40%, by weight of the reaction mixture, of water.

The term "homogeneous aqueous reaction mixture" used herein refers to a substantially solubilised system in which substantially no phase segregation occurs prior to the polymerisation reaction. For example, an emulsion, microemulsion or phase-separated mixture in which a polymerisation reaction later occurs is not a homogeneous aqueous reaction mixture as understood for the purpose of the present invention. Where a reaction mixture includes hydrophobic components, special measures will therefore be required, to achieve homogeneity, as described in more detail herein.

#### Surface Characteristics of the Polymerised Materials

It is a consequence of this invention that the phase separated polymerised material contains at least at its surface relatively hydrophobic regions, which enable the polymer to function as a pressure sensitive adhesive, and substantially hydrophilic regions, which enable the surface active agent to function in an aqueous environment at the interface between the polymer and mammalian skin. When the polymer is placed in contact with skin, the nature and quantity of surface active agent are chosen to bring about the removal of natural or synthetic hydrophobic material, such as skin lipid or skin crème, from the skin surface without adversely diminishing the work of adhesion between the hydrophobic domains and the skin surface. In as much as both the polymeric adhesive formed in this invention and the skin with which it is contacted are deformable under conditions of normal use, an equilibrium interfacial situation is reached in which some spatial exchange of hydrophobic regions and hydrophobic regions will take place on the skin surface.

The phase separated polymerised surface material is found to include predominantly well defined hydrophobic phases embedded in a hydrophilic matrix in which the water is predominantly contained. The hydrophobic phases are generally of elongated form, with a transverse dimension above the wavelength of light (e.g. about 0.5 to about 100 microns). They may therefore be visualised under a light microscope on a sample stained with a dye which binds preferentially to the hydrophobic phase.

The surface morphology of the elongate hydrophobic phases can vary widely. Without wishing to be bound by theory, it is believed that variations in the surface tension at the hydrophobic/hydrophilic interface as the polymerisation reaction proceeds can cause the morphologies to vary in the final polymer. This surface tension can be affected by the nature and amount of both the reactive solvent bridge and the surfactant, and by other factors.

Thus, it is possible for the elongate hydrophobic phases at the surface of the polymerised material to congregate in a clustered, or alternatively a relatively open, arrangement. The hydrophobic phase visualised microscopically may, for example, appear as discontinuous linear and/or branched strands, or closed loops, embedded in the hydrophilic matrix.

The polymerised material is typically non-bicontinuous. At least one of the hydrophobic and hydrophilic phases exists as discrete regions within the polymerised material, and both phases do not simultaneously extend across the polymerised material (bicontinuity).

In preparing adhesive compositions in accordance with the invention, the ingredients will usually be mixed to provide a homogeneous reaction mixture in the form of an initial pre-gel aqueous based liquid formulation, and this is then converted into a gel by a free radical polymerisation reaction. This may be achieved for example using conventional thermal initiators and/or photoinitiators or by ionizing radiation. Photoinitiation is a preferred method and will usually be applied by subjecting the pre-gel reaction mixture containing an appropriate photoinitiation agent to UV light after it has been spread or coated as a layer on siliconised release paper or other solid substrate. The incident UV intensity, at a wavelength in the range from 240 to 420nm, is ideally substantially 40mW/cm<sup>2</sup>.

The processing will generally be carried out in a controlled manner involving a precise predetermined sequence of mixing and thermal treatment or history.

5 The UV irradiation time scale should ideally be less than 60 seconds, and preferably less than 10 seconds to form a gel with better than 95% conversion of the monomers and for conversion better than 99.95% exposure to UV light less than 60 seconds and preferably less than 40 seconds is preferred. Those skilled in the art will appreciate that the extent of irradiation will be dependent on the thickness of the reaction mixture, concentration of photoinitiator and nature of  
10 substrate on to which the reaction mixture is coated and the source of UV.

These timings are for medium pressure mercury arc lamps as the source of UV operating at 100 W/cm. The intensity of UV @ 254nm and 313nm reaching the surface of the substrate is approximately  $150\mu\text{W}/\text{cm}^2$  and  $750\mu\text{W}/\text{cm}^2$ . For a  
15 given lamp UV intensity in a function of the operating power and distance of the reaction mixture from the UV source.

In order to minimize and preferably eliminate the presence of any residual monomers it is important to ensure that the reaction is complete. This is  
20 dependent upon a number of factors such as the substrate onto which the adhesive is applied, the type and intensity of the ultra violet light and the number of ultra violet light passes. Preferably the conversion of the hydrophilic monomers present such as NaAMPS should be 98%, preferably 99.0% most preferably 99.9% so that the amount of monomer within the adhesive is 4600 microg/g or less, preferably 2300 microg/g or less, most preferably 230 microg/g or less.  
25 Similarly, the conversion of the hydrophobic monomers present such as NNDMA should be 99%, preferably 99.9%, most preferably 99.99% so that the amount of monomer present in the adhesive is 2200 microg/g or less, preferably 220 microg/g or less, most preferably 22microg/g or less.

30 The adhesive is provided, typically on at least a portion of the wearer facing surface of the article, as a layer having a thickness or calliper C that is preferably constant, or that alternatively can vary over the surface of application of the adhesive.



When considering particularly the removal phase of an adhesive composition for attachment to the skin of a wearer, it is commonly recognised that good conditions of removal, i.e. at a frequency of about 100 rad/sec, of the adhesive applied to at least part of the wearer facing surface of the article, are achieved when the adhesive can be easily removed from the skin, and particularly from the bodily hair that may be located on this area of the skin, where the article contacts the body, without causing pain to the wearer, therefore without adhering too hard upon removal, to the skin and the hair of the wearer. Moreover, a good removal implies that the adhesive does not leave residues on the skin or on the hair. The relationship between the thickness or calliper C measured in millimetres (mm) of the layer of the adhesive typically onto at least part of the wearer's facing surface of the absorbent article and the viscous modulus  $G''_{25}$  at 25°C at about 100 rad/sec of the adhesive gives an indication of painless and easy removal of the adhesive from the skin.

15

Without being bound to any theory, it is believed that for higher values of  $G''_{25}$  at 100 rad/sec, which overall correspond to a higher adhesiveness of the composition, a thicker calliper or thickness C of the adhesive layer is needed so that the energy applied for the removal is more evenly distributed within the mass of the adhesive, and is therefore transferred smoothly to the skin, so avoiding peaks of energy that typically cause the pain sensation to the wearer. In other words, thinner layers of the adhesive necessitate an adhesive with a lower  $G''_{25}$  at 100 rad/sec to achieve a reduced pain sensation upon removal of the article.

20

According to the present invention, the adhesive is preferably provided as a layer having a thickness C such that the viscous modulus  $G''_{25}$  (100 rad/sec) and the thickness C of the adhesive layer satisfy the following empirical equation:

25

$$G''_{25} \leq [(7.00 + C) \times 3000] \text{ Pa}$$

30

and preferably the following empirical equation:

$$G''_{25} \leq [(5.50 + C) \times 1700] \text{ Pa}$$

35

While in a preferred embodiment of the present invention the thickness C of the adhesive layer is constant, such an adhesive layer can also have different

thicknesses in different portions of the wearer facing surface where it is applied, provided that the above mentioned relationship between  $C$  and  $G''_{25}$  is in any case satisfied in each portion.

5 In order to evaluate the effect of the thickness  $C$  of the adhesive layer in its relationship with the viscous modulus  $G''_{25}$  (100 rad/sec) of the adhesive of the present invention on the removal of the adhesive used for the attachment of a article to the skin of a wearer, a Removal Pain Grade Test has been developed. In this test the adhesion of standard substrates, on which the same adhesive has  
10 been provided in layers having different thicknesses, on the skin of the forearm of members of a sensory panel is achieved, and upon successive removal the pain is evaluated in terms of pain grade as described herein after.

#### Description of the disposable absorbent article

15 Absorbent articles in which the adhesive according to the present invention can be used, can be made by any of the ways usual in the art. The application of the adhesive to the wearer facing surface, typically the topsheet surface of an absorbent article should not cause major problems to those skilled in the art since it can be provided by any well known techniques commonly used to apply  
20 adhesives. Most preferably the adhesive is provided in a pattern of small incremental areas such as dots or similar.

The adhesive is applied on at least portion of the wearer facing surface of  
25 disposable absorbent articles in a layer having a thickness or caliper that is preferably constant, or that alternatively can vary over the surface interested by the application of the adhesive. The adhesive can be applied to the wearer facing surface of the article by any means known in the art such as slot coating, spiral or bead application or printing. Typically the adhesive is applied at a basis weight  
30 of from  $20\text{g/m}^2$  to  $2500\text{g/m}^2$ , preferably from  $500\text{g/m}^2$  to  $2000\text{g/m}^2$ , most preferably from  $700\text{g/m}^2$  to  $1500\text{g/m}^2$  depending in the end use envisioned.

If possible, the article also provides breathability by being at least water vapour permeable, preferably air permeable to prevent stuffiness. Breathability,  
35 if not supported by the adhesive as such, can be limited to the area of the article where no adhesive is applied.

The adhesive on an article is preferably protected prior to use. This protection can be provided by a release liner such as a siliconised or surfactant treated paper, providing easy release for the selected adhesive.

5

This invention can be used beneficially on disposable absorbent articles which are applied directly to the skin of a user. The article usually exhibits absorbency for bodily fluids, the protection of the user's garments from soiling, is comfortable to the user, and is easy to produce and to package. The disposable  
10 absorbent article is described below by reference to a sanitary napkin or catamenial, however diapers, panty liners, adult incontinence articles, tampons or perspiration pads are also included under the term disposable absorbent articles. The term "sanitary napkin", as used herein, refers to an article which is worn by females adjacent to the pudendal region and which is intended to absorb  
15 and contain the various body fluids which are discharged from the body (e.g., vaginal discharges, menses, and/or urine) and which is intended to be discarded after a single use. A disposable absorbent article is preferably thin, more preferably between 1 and 5 mm thick and either substantially flat prior to use or in a preshaped form.

20

The terms "joined" or "affixed", as used herein, encompasses configurations whereby a first member is directly connected to a second member and configurations whereby a first member is indirectly connected to a second member by connecting the first member to intermediate members which in turn  
25 are connected to the second member.

The sanitary napkin has two main surfaces, a body contacting or wearer facing surface on which the adhesive is applied and a garment facing or contacting surface. In a one preferred embodiment a sanitary napkin of the  
30 present invention comprises a liquid pervious topsheet, a liquid impervious backsheet joined to the topsheet, and an absorbent core intermediate the topsheet and the backsheet. In an alternative embodiment, the sanitary napkin or panty liner may utilize the adhesive to absorb quantities of liquid up to amounts of about 10g, such that a separate core and topsheet are not required. Such  
35 products preferably have a backsheet as described below.

The topsheet is compliant, soft feeling, and non-irritating to the wearer's skin. The topsheet also can have elastic characteristics allowing it to be stretched in one or two directions in portions of the topsheet or throughout its extension. Further, the topsheet is fluid pervious permitting fluids (e.g., menses and/or urine) to readily penetrate through its thickness.

Preferred topsheets for use in the present invention are typically selected from high loft nonwoven topsheets and apertured formed film topsheets. Apertured formed films are especially preferred for the topsheets because they are pervious to body exudates and yet non absorbent and have a reduced tendency to allow fluids to pass back through and rewet the wearer's skin. Thus, the surface of the formed film that is in contact with the wearer remains dry, thereby reducing body soiling and creating a more comfortable feel for the wearer. Suitable formed films are described in U.S. Patent 3,929,135; U.S. Patent 4,324,246; U.S. Patent 4,342,314; U.S. Patent 4,463,045; and U.S. Patent 5,006,394. Particularly preferred micro apertured formed film topsheets are disclosed in U.S. Patent 4,609,518 and U.S. Patent 4,629,643. A preferred topsheet for the present invention comprises the formed film described in one or more of the above patents and marketed on sanitary napkins by The Procter & Gamble Company of Cincinnati, Ohio as "DRI-WEAVE".

Adhesives are most suitably used on topsheets having not a homogeneous distribution of liquid passage ways but only a portion of the topsheet comprising liquid passage ways oriented such that they result in a centrally permeable and peripherally impermeable topsheet for liquids.

Another alternative are so called hybrid topsheets which incorporate fibrous and film like structures particularly useful embodiments of such hybrid topsheets are disclosed in PCT publications WO 93/09744; WO 93/11725 or WO 93/11726.

When referring to the topsheet a multi layer structure or a mono layer structure is contemplated. The hybrid topsheet mentioned above is such a multi layer design but other multi layer topsheets such as primary and secondary topsheet designs are also considered.

The absorbent core also can comprise multiple layers and provides fluid storage and distribution function.

5       Positioned in fluid communication with, and typically underlying the topsheet is the absorbent core. The core can comprise any usual absorbent material or combinations thereof. It preferably comprises absorbent gelling materials usually referred to as "hydrogel", "superabsorbent", "hydrocolloid" materials in combination with suitable carriers.

10       Suitable absorbent gelling materials for use herein will most often comprise a substantially water-insoluble, slightly cross-linked, partially neutralised, polymeric gelling material. This material forms a hydrogel upon contact with water. Such polymer materials can be prepared from polymerizable, unsaturated, acid-containing monomers, such as acrylic acid, which are well known in the art.

15       Suitable carriers include materials which are conventionally utilised in absorbent structures such as natural, modified or synthetic fibers, particularly modified or non-modified cellulose fibers, in the form of fluff and/or tissues. Suitable carriers can be used together with the absorbent gelling material,  
20       however, they can also be used alone or in combinations. Most preferred are tissue or tissue laminates in the context of sanitary napkins/panty liners.

25       An embodiment of the core, particularly useful in the application of the present invention, comprises a double layer tissue laminate formed by folding the tissue onto itself. These layers can be joined to each other. Absorbent gelling material or other optional material can be comprised between the layers.

30       The absorbent core can include optional components normally present in absorbent webs such as odor control agents, in particular suitable zeolites.

35       The backsheet primarily prevents the exudates absorbed and contained in the absorbent core from wetting articles that contact the absorbent product such as underpants, pants, pyjamas and undergarments. The backsheet is preferably impervious to liquids (e.g. menses and/or urine) and usually manufactured from a thin plastic film.

The backsheet typically extends across the whole of the absorbent core and can extend onto and form part of the topsheet by folding around the absorbent core. Thereby a topsheet configuration as disclosed in US 4,342,314, column 16, lines 47 - 62 can be achieved without the requirement to selectively aperture the topsheet.

Preferably, the backsheet also provides breathability to the absorbent article by being at least water vapour permeable, preferably air permeable. The backsheet can be a laminate material e.g. of a combination of microporous film and/or non-woven material, and/or apertured formed film. Breathability if desired can be limited to the periphery or the center of the backsheet or it can be across the whole backsheet.

According to the present invention the adhesive as described herein may also find application to attach other articles to the skin. The adhesives may for example find utility to adhere functional articles which adhere to the skin such as cosmetic or pharmaceutical delivery articles which provide a substance to the skin such as skin treatment substances, cream, lotions, hormones, vitamins, deodorants, drugs; cosmetic or pharmaceutical delivery articles provide a substance to emanate away from the skin such as insecticides, inhalation drugs, perfumes and; functional articles which are not necessarily attached to the skin, but which require a high residence time on the skin such as decorative cosmetics, (lipstick, eye shadow, stage make-up) and cleaning articles (hand cleaners, face masks and hygienic pore cleansers). Such articles are preferably non-absorbent for bodily liquids.

The adhesive may also in addition find application to attach articles to the skin such as protective articles such as genital-, knee- or elbow-protectors or bandages; clothing such as bras, surgical gowns, or parts of garments during fitting at a tailor; nasal plasters; prosthesis such as breast replacements or wigs; cold wraps e.g. to provide pain relief from bruises and to reduce swelling; thermal wraps comprising thermal cells as disclosed for example in WO 97/36968 and WO 97/49361 to provide relief of temporary and chronic pain such as neck wraps as disclosed in for example US 5 728 146, knee wraps exemplified in WO 97/01311, and back wraps as disclosed for example in US 5 741 318; hearing aids; protective face masks (for the reduction or prevention of inhalation

of noxious substances); ornamental articles such as jewelry, earrings, guises, tattoos; goggles or other eye wear; ostomy devices, tapes, bandages, dressings of general utility, wound healing and wound management devices; and biomedical skin electrodes such as ECG, EMG, EEG, TENS electrosurgery, 5 defibrillation, EMS and electrodes for facial/beauty applications; and fixation products and/or devices intended to affix patient catheters, tubing leadwires cables etc.

### Removal Pain Grade Test

10

The Removal Pain Grade Test is utilized to evaluate the pain during removal from the skin of a wearer of a sample provided with a layer of a adhesive and previously attached to the wearer's skin. The test specifically evaluates the pain upon removal of each sample as compared to the pain obtained by 15 removing a reference sample constituted by a commercial strong medical plaster.

#### Sample preparation.

The test is performed on rectangular samples 60x20 mm made of a 20 polyester film 23  $\mu$ m thick, such as that sold by Effegidi S.p.A. of Colorno (Parma, Italy), provided on one side with a continuous layer of the adhesive having the selected thickness. The reference sample is a 60x20 mm sample of an adhesive non woven fabric available from Beiersdorf A.G. Hamburg, Germany under the Tradename Fixomull stretch.

25

#### Test method.

A panel of six graders is selected for the test. The test is performed in a climatically controlled laboratory maintained at a temperature of 23° C and a 30 Relative Humidity of 50%. No special treatment of the wearer's skin is required beyond normal cleaning/washing with water and soap. The skin is then allowed to dry for at least two hours before the test to allow the skin to reach equilibrium with th room conditions. Different adhesive are evaluated in the test in comparison with the reference sample R. Each sample is applied by hand by an 35 operator to the inner part of the grader's forearm, being centred between the wrist and the elbow, with the short side of the sample aligned with the length of

the arm. The operator exerts on each sample with the palm of the hand the same pressure that is typically applied to cause a medical plaster to adhere to the skin. Each sample is worn for the prescribed time, and then it is removed from the grader's skin by the operator with a slow and smooth pull.

5

Four series of one reference sample R and the test samples are each applied, worn and then removed from the wearer's skin; each sample is worn for one minute, with a 5 minute wait between two subsequent samples of the same series, and a 15 minute wait between two different subsequent series. The reference sample R is always applied, worn and removed as the first sample of its respective series. The sequence of application/wear/removal of the test samples in each of the first three series is random, provided that no repetition in each series is allowed, and that no sequence is repeated in the first three series. In the fourth series one of the test samples is tested twice, the reference R always being the first one. Overall each sample has to be tested an equal number of times (24 times).

10

15

The graders were asked to evaluate each sample using a pain scale ranging from 0 to 10, where 0 corresponds to no pain and 10 corresponds to the pain upon removal of the reference sample R. The pain values for each sample were obtained as a mean of 24 observations.

20

The results collected from the test were analysed by a statistical analysis program "Comparison of Population Means - Paired Samples", that showed that the differences between the pain values of the samples are statistically significant.

25

#### Peel adhesion method

30

This is a quantitative method to determine the average peel force required to remove a skin at a specified peel angle and speed.

#### Equipment

Scissors	Convenient source
Standard ruler	Convenient source



Steel Roller	5.0kg Mass. 13cm in diameter and 4.5cm in width covered with 0.5mm thick rubber.
Polyester Film	PET 23 $\mu$ available from EFFEGIDI S.p.A., 43052 Colorno, Italy.
Transfer Adhesive	3M 1524 available from 3M Italia S.p.a. ,20090 Segrate Italy
Stop watch	Convenient source
Tensile Tester	Instron mod.: 6021( or equivalent)

### Test procedure

#### A) Tensile Tester Peel Settings:

5	Load cell	10N
	Test Speed	1000 mm/min
	Clamp to Clamp distance	25 mm
	Pre Loading	0.2N
	Test Path "LM"	50mm
10	Measure variable	F average (N) in "LM"

#### B) Skin Condition and Preparation

The sample is peel from the forearm. There are 3 conditions of the skin that are tested:

- 15 1) Dry: The forearm is untreated and not wiped prior to test or between repetitions.
- 20 2) Wet: To one cotton disk (Demak'up diameter 5.5cm, weight about 0.6g), 3ml of distilled water is added. Next the disk is then wiped with a light pressure 3 times over the test area on the forearm. (The test area of the forearm is a rectangle approximately 2cm wider and longer than the adhesive area).

#### C) Sample preparation

- 25 1. Allow the samples to adjust to conditioned room ( $23 \pm 2^\circ$  Celsius and  $50 \pm 2\%RH$ ) for about 1hr.

2. Prepare rectangular adhesive samples 260mm  $\pm$ 2 length and 20mm  $\pm$ 2 wide.
3. Attach on the sample surface the polyester film (using the transfer adhesive to attach the polyester to the substrate surface).
4. Each test specimen should be prepared individually and tested immediately.
5. Remove the release paper from the adhesive without touching it. Attach one end to the skin (see section B).
6. Roll the Steel Roller for 160mm along the adhesive strip, once in each direction.

D) Test Environment

There are 2 environments the adhesive can be tested in:

- 1) Conditioned Room as described in C1.
- 2) Wet Environment. Here, after step C4, the specimen is taken and put in a humidity controlled oven for 3 hours at 85degC. It is then taken out and steps C5, C6 are carried out.

E) Execution

1 minute after Step C6, take the free end of the specimen (approx. 100mm long) and insert it in the upper end of the adhesion testing machine. Ensure the specimen is at a 90 degree angle to the forearm. Start the testing machine.

F) Report

Report the average of the peel strength of 5 tests. The single values are the base to calculate the standard deviation between the samples.

Residual Monomer Test Method

### Test Sample

1 gram of a hydrogel sample is taken and emersed in 100ml 0.9% saline water.

5 The sample is left in the saline at 40degC for 24 hours.

An aliquot of the liquid is diluted and analysed by electrospray LC/MS/MS.

### Calibration Sample

10 1 gram of reference monomers (eg NaAmps) are dissolved in 100ml 0.9% saline water.

An aliquot of the liquid is diluted and analysed by electrospray LC/MS/MS.

### Evaluation

15 The concentration of the test and calibration sample are determined by linear regression analysis using a software package such as VG Mass Lynx.

### Adhesive preparation

20 The following is the general description to prepare 20 kg of the adhesive at room temperature and pressure. 250 ml of triethanolamine is placed in a 80 l plastic beaker and stirred. N,N,-dimethylacrylate (NNDMA) is added to the beaker and stirred. Glycerol is then added and the entire mixture is stirred for 5 minutes. Sodium 2 acrylamide 2 methyl propane sulphonic acid (NaAMPS) is then added and the mixture stirred for 5 minutes. The crosslinker is then added  
25 and the mixture is again stirred for at least 30 minutes. The mixture is then extruded onto a substrate material prior to UV curing. The UV curing consists of 2 - 4 passes under a bank of 3 UV lights.

30 The reference material is prepared as above, except that the NNDMA is replaced by acrylic (3 sulphopropyl) ester acid SPA and the mixture is stirred for 24 hrs. at 53°C prior to UV curing.

All formulations d1etailed below were coated onto a polyurethane foam (EV 1700X from Caligen) at a coat weight of 0.8 to 1.6 kg per square meter and cured

by expose to ultraviolet radiation emitted from a medium pressure mercury are lamp operating at 100w/cm power for 10 seconds.

#### Adhesive Composition

5

<b>Adhesive</b>	<b>Reference</b>	<b>1</b>	<b>2</b>	<b>3</b>
NaAMPS (58%)	37	40	32.5	32.5
NNDMA	-	23.5	18	18
Glycerol	33	30	45	40
SPA	15	-	-	-
Vinyl acetate	10	-	-	-
Photoinitiator	0.03	0.07	0.23	0.23
Crosslinker	0.11	0.13	0.05	0.07
No. of UV passes	4	1	3	3

#### Results

<b>Adhesive</b>	<b>Reference</b>	<b>1</b>	<b>2</b>	<b>3</b>
$P_I$ (N/cm)	2.5	2.55	2.70	0.95
$P_F$ (N/cm)	1.43	2.10	2.70	1.81
P (Water absorbency of 20%, N/cm)	0.43	-	1.88	1.13
$G'_{37}$ (dynes/cm <sup>2</sup> )	90712	187680	171330	181340
$G''_{37}$ (dynes/cm <sup>2</sup> )	59338	104750	119400	124660

## 5 WHAT IS CLAIMED IS:

1. A disposable absorbent article comprising a wearing facing surface and a garment facing surface, said wearing facing surface comprising at least one portion comprising an adhesive, characterised in that said adhesive has an  
10 initial peel strength ( $P_I$ ) and a final peel strength ( $P_F$ ) after exposure to water as defined in the test method herein wherein the ratio of  $P_I$  to  $P_F$  is from 2:1 to 2:4 and a water absorption capacity as defined in the test herein of at least 3% by weight of said adhesive.
- 15 2. An absorbent article according to claim 1, wherein the ratio of said initial peel strength ( $P_I$ ) to said final peel strength ( $P_F$ ) is from 2:1.25 to 2:2.
3. An absorbent article according to any one of the preceding claims, wherein said initial peel strength ( $P_I$ ) of said adhesive is from 0.1N/cm to 5.0N/cm.
- 20 4. An absorbent article according to any one of the preceding claims, wherein said initial peel strength ( $P_I$ ) of said adhesive is from 0.5N/cm to 3.0N/cm.
5. An absorbent article according to any one of the preceding claims, wherein  
25 adhesive is provided as a layer having a thickness  $C$  measured in millimetres (mm),

said adhesive having a viscous modulus at a temperature of 25°C (77°F),  $G''_{25}$ ,

30 wherein said viscous modulus  $G''_{25}$  (100 rad/sec) and said thickness  $C$  of said adhesive satisfy the following equation:

$$G''_{25} \leq [(7.00 + C) \times 3000] \text{ Pa.}$$

6. An absorbent article (10) according to claim 5 characterized in that said viscous modulus  $G''_{25}$  (100 rad/sec) and said thickness C satisfy the following equation:

5 
$$G''_{25} \leq [(5.50 + C) \times 1700] \text{ Pa.}$$

7. An absorbent article according to claims 5 or 6, characterized in that said adhesive has an elastic modulus at a temperature of 37°C (100°F),  $G'_{37}$ , and having a viscous modulus at a temperature of 37°C (100°F),  $G''_{37}$ , and is selected to have;
- 10 a)  $G'_{37}$  (1 rad/sec) in the range 500 Pa to 20000 Pa, preferably 700 Pa to 15000 Pa, most preferably 1000 Pa to 10000 Pa;
- 15 b)  $G''_{37}$  (1 rad/sec) in the range 100 Pa to 15000 Pa, preferably 100 Pa to 10000 Pa, most preferably 300 Pa to 5000 Pa; and
- c) the ratio  $G'_{37}$  (1 rad/sec) /  $G''_{37}$  (1 rad/sec) in the range 1 to 30.
- 20 8. An absorbent article according to any one of the preceding claims, wherein said adhesive is a substantially water insoluble pressure sensitive adhesive comprising a polymer which forms a 3-dimensional matrix, and comprising less than 10 %, preferably less than 5 % by weight of said adhesive of hydrocolloid particles.
- 25 9. An absorbent article according to claim 8, wherein said adhesive comprises
- a) a polymer selected from acrylics, sulphonated polymers, vinyl alcohols, vinyl pyrrolidine, polyethylene oxide or mixtures thereof and
- 30 b) a plasticizer selected from polyhydric alcohols, polyethylene glycols, sorbitol, water or mixtures thereof.
- 35 10. An absorbent article according to claim 8, wherein said adhesive is a hydrophilic-hydrophobic mixed phase adhesive.

11. An absorbent article according to any one of the preceding claims, wherein said adhesive is formed by polymerizing a homogeneous aqueous reaction mixture comprising from 5 to 50% by weight of the reaction mixture of a hydrophilic monomer, from 10 to 50% by weight of the reaction mixture of a plasticiser (other than water) up to 50% by weight of the reaction mixture of a nonionic monomer and up to 40% by weight of the reaction mixture of water.
12. An absorbent article according to any one of the preceding claims, wherein said wearer facing surface of said article comprises at least one non-adhesive portion.
13. An absorbent article according to any one of the preceding claims, characterized in that said adhesive is provided as a continuous layer.
14. An absorbent article according to any one of the preceding claims, wherein said adhesive is applied to said wearer facing surface by slot coating.
15. An absorbent article according to any one of the preceding claims, wherein said article comprises a backsheet and said backsheet is in direct contact with said adhesive.
16. An absorbent article according to any one of the preceding claims 1 to 14, wherein said article comprises a topsheet, a backsheet and an absorbent core located inbetween said topsheet and said backsheet.





# INTERNATIONAL SEARCH REPORT

Intern      nal Application No  
PCT/US 00/02706

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 7    A61L15/58    A61F13/15    A61F5/44

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
IPC 7    A61L    A61F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, WPI Data

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0 850 649 A (PROCTER & GAMBLE) 1 July 1998 (1998-07-01) page 4, line 33-49 page 7, line 20-58; claim 4	1-11, 15, 16
Y	WO 99 00092 A (ACCHIOLI VINCENZO D ;PALUMBO GIANFRANCO (DE); PROCTER & GAMBLE (US) 7 January 1999 (1999-01-07) abstract page 1, paragraph 2 page 13, paragraph 1 page 14, paragraph 3	1-11, 15, 16

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

\* Special categories of cited documents:

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \*&\* document member of the same patent family

Date of the actual completion of the international search

21 June 2000

Date of mailing of the international search report

28/06/2000

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Böhm, I

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 00/02706

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	GB 2 115 431 A (VALLEYLAB INC) 7 September 1983 (1983-09-07) cited in the application abstract page 4, line 16-21 page 7, line 3,4 claims 1,7	1-11,15, 16
A	EP 0 554 106 A (ADHESIVES RES INC) 4 August 1993 (1993-08-04) abstract page 3, line 5-30 page 4, line 3-14 claim 1	1-10
A	US 5 015 244 A (CROSS DAVID E) 14 May 1991 (1991-05-14) abstract column 1, line 13-24 column 2, line 55-61 column 3, line 24-30 column 3, line 24-30	1-10
A	EP 0 850 625 A (PROCTER & GAMBLE) 1 July 1998 (1998-07-01) page 3, line 13-38 page 4, line 55 -page 5, line 3 page 10, line 42-52 page 11, line 18-33 page 10, line 42-52 -page 11, line 18-33	1-10

## INTERNATIONAL SEARCH REPORT

International Application No. PCT/US 00 02706

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box I.2

Claims Nos.: 1-7

Present claims 1-7 relate to a product defined (inter alia)  
by reference to the following parameter(s):

P1, P2 : the initial peel strength ( $P(i)$ ), the final peel strength ( $P(f)$ )  
;  
the ratio of  $P(i)$  to  $P(f)$  from 2:1 to 2:4

P3 : water absorption capacity > 3%

P4 : elastic modulus  $G'(37)$

P5 : viscous modulus  $G''(37)$

The use of these parameters in the present context is considered to lead to a lack of clarity within the meaning of Article 6 PCT. It is impossible to compare the parameters the applicant has chosen to employ with what is set out in the prior art. The lack of clarity is such as to render a meaningful complete search impossible. Consequently, the search of claims 1-7 has been restricted to choose claimed devices in which the adhesive is that according to claim 8.

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 00/02706

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0850649 A	01-07-1998	EP 0850619 A	01-07-1998
		EP 0853934 A	22-07-1998
		AU 5385398 A	17-07-1998
		AU 5619598 A	17-07-1998
		AU 5801498 A	17-07-1998
		CN 1246063 A	01-03-2000
		CN 1246066 A	01-03-2000
		CN 1246067 A	01-03-2000
		EP 0948364 A	13-10-1999
		EP 0946213 A	06-10-1999
		EP 0948368 A	13-10-1999
		WO 9828015 A	02-07-1998
		WO 9828018 A	02-07-1998
		WO 9828022 A	02-07-1998
		AU 5531598 A	17-07-1998
		AU 5612598 A	17-07-1998
		AU 5612698 A	17-07-1998
		AU 5612998 A	17-07-1998
		AU 5619698 A	17-07-1998
		AU 5619798 A	17-07-1998
		AU 5708898 A	17-07-1998
		AU 5709198 A	17-07-1998
		AU 5709498 A	17-07-1998
		AU 5801598 A	17-07-1998
		CN 1246060 A	01-03-2000
		CN 1246061 A	01-03-2000
		CN 1246062 A	01-03-2000
		CN 1246044 A	01-03-2000
		CN 1246064 A	01-03-2000
		CN 1246065 A	01-03-2000
		CN 1246068 A	01-03-2000
		EP 0850624 A	01-07-1998
		EP 0850628 A	01-07-1998
		EP 0852149 A	08-07-1998
		EP 0855190 A	29-07-1998
		EP 0957871 A	24-11-1999
		EP 0948365 A	13-10-1999
		EP 0968007 A	05-01-2000
		EP 0946214 A	06-10-1999
		EP 0946215 A	06-10-1999
		EP 1006973 A	14-06-2000
		EP 0948366 A	13-10-1999
		EP 0948367 A	13-10-1999
		EP 0948369 A	13-10-1999
		NO 993054 A	23-08-1999
		NO 993060 A	23-08-1999
		WO 9827909 A	02-07-1998
		WO 9828014 A	02-07-1998
		WO 9827914 A	02-07-1998
		WO 9828016 A	02-07-1998
WO 9900092 A	07-01-1999	EP 0887061 A	30-12-1998
		EP 0887062 A	30-12-1998
		EP 0887063 A	30-12-1998
		AU 8172198 A	19-01-1999
		AU 8172298 A	19-01-1999
		AU 8172398 A	19-01-1999
		AU 8172498 A	19-01-1999

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 00/02706

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 9900092 A		AU 8172698 A	19-01-1999
		AU 8268798 A	19-01-1999
		AU 8268998 A	19-01-1999
		AU 8269098 A	19-01-1999
		AU 8269198 A	19-01-1999
		EP 0991388 A	12-04-2000
		EP 0991389 A	12-04-2000
		EP 0991390 A	12-04-2000
		EP 0991391 A	12-04-2000
		EP 0991392 A	12-04-2000
		EP 0991393 A	12-04-2000
		EP 0991394 A	12-04-2000
		EP 0993292 A	19-04-2000
		EP 0993293 A	19-04-2000
		WO 9900084 A	07-01-1999
		WO 9900085 A	07-01-1999
		WO 9900086 A	07-01-1999
		WO 9900087 A	07-01-1999
		WO 9900088 A	07-01-1999
		WO 9900089 A	07-01-1999
		WO 9900090 A	07-01-1999
		WO 9900091 A	07-01-1999
		ZA 9805620 A	28-12-1998
		ZA 9805617 A	13-04-1999
		ZA 9805623 A	13-04-1999
GB 2115431 A	07-09-1983	AU 536939 B	31-05-1984
		AU 1058383 A	29-09-1983
		AU 1230288 A	26-05-1988
		CA 1218954 A	10-03-1987
		DE 3305473 A	02-02-1984
		FR 2522006 A	26-08-1983
		JP 1695909 C	28-09-1992
		JP 58162681 A	27-09-1983
		JP 63065235 B	15-12-1988
		JP 1787645 C	10-09-1993
		JP 2211145 A	22-08-1990
		JP 4078310 B	10-12-1992
		US 4699146 A	13-10-1987
		US 4750482 A	14-06-1988
EP 0554106 A	04-08-1993	US 5352516 A	04-10-1994
		AT 136576 T	15-04-1996
		CA 2088457 A,C	01-08-1993
		DE 69302107 D	15-05-1996
		DE 69302107 T	29-08-1996
US 5015244 A	14-05-1991	DE 3935824 A	10-05-1990
		DE 3935825 A	10-05-1990
		DK 554889 A	09-05-1990
		DK 554989 A	09-05-1990
		ES 2017318 A	16-01-1991
		FR 2638633 A	11-05-1990
		FR 2638634 A	11-05-1990
		GB 2226761 A,B	11-07-1990
		JP 2180255 A	13-07-1990
		JP 2172470 A	04-07-1990

# INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

PCT/US 00/02706

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0850625 A	01-07-1998	EP 0850620 A	01-07-1998
		AU 5613298 A	17-07-1998
		EP 1006972 A	14-06-2000
		WO 9827917 A	02-07-1998
		AU 5531398 A	17-07-1998
		EP 0946212 A	06-10-1999
		WO 9828017 A	02-07-1998